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COMPRESSION TESTING OF CONTINUOUS P-100 FIBER REINFORCED GLASS MATRIX COMPOSITE TUBES

Prepared by

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INTERIM REPORT

Contract N00014-89-C-0046

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Four glass matrix composite tubes reinforced with continuous P-100 carbon fiber were fabricated at United Technologies Research Center on an Office of Naval Research program using hot isostatic pressing. These tubes were supplied to Southern Research Institute for compression testing as part of an NSWC program to evaluate composite materials for satellite applications. The resulting properties of the four tubes exceeded the NSWC program requirements and were comparable to those of carbon-carbon tubes that had been previously evaluated at Southern Research. This equivalent mechanical performance together with the demonstrated atomic oxygen resistance, thermal damage tolerance, temperature capability to 1200°C, and ease of fabrication suggests that carbon fiber reinforced glass matrix composites may offer acceptable performance for structural satellite applications.

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COMPRESSION TESTING OF CONTINUOUS P-100 FIBER REINFORCED GLASS MATRIX COMPOSITE TUBES

INTERIM REPORT

Office of Naval Research
Contract N00014-89-C-0046

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INTRODUCTION

United Technologies Research Center (UTRC) currently has a program with the Office of Naval Research funded by the IS&T branch of SDIO to develop carbon fiber reinforced glass (C/Gl) matrix composites for space-based applications. C/Gl composites have been selected for potential space satellite structural applications because of their high stiffness and strength, low density, high temperature capability, space environmental durability, and relative ease of fabrication. Many of these characteristics have been demonstrated in recent years on the ONR program as well as other related activities. For example, resistance to atomic oxygen (AO) attack has been demonstrated through the analysis of C/Gl samples that were exposed to the space environment for nearly 6 years on the Long Duration Exposure Facility (LDEF) [1]. In another aspect of survivability, thermal damage tolerance of C/Gl composites has been demonstrated through the testing of flat panels at Oak Ridge National Lab [2]. High temperature strength retention of C/Gl composites has been demonstrated up to 600°C [3] and 1200°C for borosilicate and glass-ceramic matrices, respectively. Also, fabrication of large 8" x 20" x 0.030" flat plates (for such applications as satellite radiator fins) has been demonstrated to be possible in single step consolidation cycles of less than 6 hours duration.

One of the tasks on the current ONR program has been to develop advanced fabrication techniques for structural components such as tubes, beams, and large thin-gage panels. During the past 18 months, continuously reinforced thin-walled tubes have been successfully fabricated using hot isostatic pressing (HIP) as the method of consolidation. High quality tubes up to 12" long with inner diameters ranging from 1" to 1.75" and wall thicknesses as low as 0.030" have been demonstrated on the program. Figure 1 shows several of these tubes as well as a right-angle bracket that have been fabricated via HIP.

Four borosilicate glass tubes reinforced with P-100 fiber (P-100/BSG composites) were fabricated on the ONR program and submitted to Southern Research Institute (SoRI) for compression testing. The compression testing was performed as part of a NSWC program to develop composite tubes for satellite applications (Satellite Applications for Carbon-Carbon II, or SACC II). Ultra-high modulus pitch-based P-100 carbon fiber was selected as the reinforcement because of the need for high specific stiffness in the SACC II applications. These composites were the first C/Gl thin-walled tubes to be evaluated for mechanical performance and looked very promising in terms of their compressive behavior.

This report summarizes the results of this testing and compares the C/Gl tube performance with that of C-C tubes tested previously on the SACC II program. In addition, the results of an analytical modeling study to predict tube performance as well as fabrication and evaluation of the P-100/BSG tubes at UTRC are discussed.

PROCEDURE

• Analytical Modeling

The mechanical performance of C/Glass composites reinforced with P-100 fiber has been previously modeled by Materials Sciences Corporation (MSC) on a Phase I SBIR program to identify potential satellite applications for C/Glass composites. The model devised by MSC, which was based on the concentric cylinder assemblage model, was able to accurately describe the mechanical behavior of unidirectional P-100 fiber reinforced glass composites. Using that experience base, MSC performed analytical modeling of the P-100/BSG composite tubes in order to determine an optimum fiber geometry that would supply high axial strength and stiffness while also providing some degree of hoop reinforcement. While a tube with all of the fibers aligned axially would provide the highest strength and stiffness, it was believed that some off-axis hoop reinforcement was necessary to make the tube easier to handle. MSC* used compression data generated on flat panels of 0° P-100/BSG as input for the model and then calculated compression properties for a thin-walled tube (1.5" ID, 0.030" wall thickness) as a function of the orientation of the off-axis plies. It was assumed that the tube would contain 40 vol% fiber and would have a fiber geometry described by the ply layup [0/+Θ/0/0/-Θ/0]. The value of Θ was allowed to vary from 0° to 30°.

Figures 2 and 3 show the results of these calculations for ultimate compressive load and compressive elastic modulus, respectively. The design goals dictated by the SACC II program are also included on the figures. The calculations indicate that even an orientation of ±30° for the off-axis plies still provides a compressive load and elastic modulus exceeding the design requirements. MSC recommended that an off-axis orientation of 20° or less would be preferred since this would provide a comfortable margin above the design goal. Inasmuch as it was desirable to maximize the amount of hoop reinforcement in the tubes, the [0/+20/0/0/-20/0] ply orientation was selected as the construction for the P-100/BSG composite tubes to be fabricated for testing at SoRI. (The calculations performed by MSC also predicted that this orientation would meet the design goals for tensile strength and thermal expansion.)

• Tube and Panel Fabrication

The C/Gl tubes consisted of a matrix of borosilicate glass reinforced with ~38-40 vol% of P-100 carbon fiber. As stated previously, each tube was constructed of six plies with an orientation of [0/+20°/0/0/-20°/0]. The 6" long tubes had an average inner diameter and wall thickness of 1.491" and 0.035", respectively, indicating an average ply thickness of ~0.006".

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* Calculations performed by Kent Buesking

Fabrication of the tubes was carried out in the following manner. Prepregged unidirectional tape was fabricated by drawing the fiber through an aqueous slurry bath containing the borosilicate glass powder and an organic binder. Each ply of the six ply composite was cut according to the desired lay-up. Unidirectional plies were straight forward to cut, while the angle plies were cut in such a fashion that when placed in the composite tube the seam would spiral down the length. This procedure prevented the existence of an axial plane of weakness in the off-axis plies. The ply dimensions and the geometry for the off-axis plies are shown in Figure 4. These dimensions were calculated based on a tube outer diameter of 1.570". Each of the plies were cut for this dimension; i.e. no correction was made for the decreasing diameter as plies are added.

After being wrapped around a mandrel, the prepreg lay-up was thermally treated to remove the organic binder prior to encapsulation in the HIP container. The encapsulated material was vacuum de-gassed at elevated temperature for 30 minutes and sealed. The HIP can, now under vacuum, was loaded into the pressure vessel of the HIP unit. The vacuum inside the can is critical and allows the can to deform into the void as external pressure is applied, thereby consolidating the prepreg material. Fabrication of the continuous fiber reinforced thin-walled tubes was facilitated though the use of a special HIP can design developed at UTRC. Consolidation was carried out at a temperature sufficient to allow flow of the glass matrix to completely densify the composite. After consolidation, the tubes were removed from the encapsulation and submitted for inspection.

Additionally, continuous fiber reinforced P-100/BSG flat panels were fabricated from identical starting materials using uniaxial hot pressing. For these samples, the prepreg tape was cut into 4" x 4" sections of the desired fiber orientation. The plies were stacked and thermally treated to remove the organic binder. The composite preform was transferred to a graphite die and hot pressed using a similar time/temperature/pressure profile to the HIP'ed material. This material was cut into test specimens for evaluation and comparison with the performance of the composites fabricated via HIP.

- **Evaluation of Tube Quality**

Several techniques were used to assess the quality of the tubes prior to compression testing. Visual inspection indicated that the outer surface of the tubes was smooth and essentially free of any defects. The inner surface of the tubes was also quite smooth, but occasionally contained small rough patches associated with the HIP encapsulation. Non-destructive evaluation (NDE) techniques used to evaluate the tubes included ultrasonic C-scan and X-ray radiography, which were performed at UTRC, as well as profilometry measurements, which were performed at SoRI after receiving the tubes. A sample of the ultrasonic data collected on two of the tubes is shown in Figure 5. The spiked-pulse reflected amplitude data shown is a

measure of attenuation of the sound wave as it passes through the material, is reflected off a metallic center support, passes through the material again, and is collected at the source. Areas blue in color indicate low sound wave amplitude, or high attenuation. This technique is useful for identifying surface or internal composite defects, which are the source for attenuation in the material.. As shown in Figure 5(a), sample 95-91 has a high concentration of defects in the gage section, while sample 92-91 appears more uniform in this area [Figure 5(b)]. Even though tube 92-91 does show low amplitude at one end, it was determined that this would not interfere with the test since this area would be encased in the grip.

In addition, ultrasonic scans were used to determine the thickness and variation in thickness by monitoring the time of flight of the sound wave in the material. The time required for a sound wave to pass through the sample, reflect off the inner wall, and return to the sensor is an indication of the part thickness. The time measurement is translated to part thickness simply by making physical measurements at known locations and assuming a 1:1 relationship of time-of-flight:part thickness. Figure 6 shows a time-of-flight ultrasonic scan of tube 94-91. The scan shows that the tube varies in wall thickness circumferentially, but is fairly constant in thickness down the length of the tube in any given location.

Profilometry measurements, performed at SoRI, were also used to measure tube wall thickness. Profilometry can continuously measure the diameters, both inner and outer, of a tube, thus allowing an accurate average wall thickness to be determined. The UTRC time-of-flight data and the SoRI profilometry data compared well with each other. For example, SoRI measured the thinnest area of tube 94-91 to be 0.025" using profilometry, while UTRC's time-of-flight measurement of the same area gave a wall thickness of 0.023".

X-ray analysis of the tubes was useful in determining the presence of high density inclusions or damaged fibers. X-ray analysis was performed by placing strips of film inside the tube and exposing the outside to X-radiation. In addition to monitoring inclusions or damaged fiber, this NDE technique allows fiber orientation to be checked. The X-ray image for a section of tube 94-91 shown in Figure 7, in which the off-axis orientation of fibers is clearly visible, indicates that the off-axis fibers spiral continuously down the length of the tube.

An overall assessment of tube quality was made based on the ultrasonic and X-ray analyses performed at UTRC. Specimen 95-91 was determined to be of the lowest quality and therefore recommended to be used as a "learning curve" sample. This specimen showed a high density of material defects in ultrasonic analysis [shown earlier in Figure 5(a)], and broken fibers in both visual and X-ray inspection. In addition, this material showed evidence of wide variations in wall thickness. UTRC believed that having a "learning curve" sample would allow SoRI to get a feel for the behavior of the C/Gl material prior to testing the other tubes. The remaining three specimens (92-91, 93-91, 94-91) all appeared to be in excellent condition and of essentially equivalent quality.

- **Compression Testing**

In order to fit the C/Gl tubes into the grips that were designed for the SACC II program, the ends of some of the tubes had to have their inner surface lightly machined (conducted at SoRI). However, care was taken to machine only the area required to allow the tubes to slip into the grips; the gage section of the tubes was not machined. Strain data was monitored using strain gages for all of the tubes except for the learning curve tube, which used clip-on extensometers.

RESULTS AND DISCUSSION

Table 1 shows the results of compression testing of the three high quality C/Gl tubes (specimen #'s 92-91, 93-91, 94-91) as measured by SoRI. Also shown are the results for the learning curve tube as well as the predicted tube properties based on the analytical calculations performed by MSC and the required tube properties as defined by NSWC. The three high quality C/Gl tubes easily exceeded the NSWC SACC II program requirements, with an average failure load more than 1000 lbs over the required load of 4500 lbs. Figure 8 shows a picture of C/Gl specimen #92-91 after compression testing. This particular specimen as well as the other samples were all observed to fail in the gage section. The jagged fracture pattern in many areas seems to follow the direction of the +20°/-20° plies. It is clear from the results in Table 1 that the learning curve tube, which was identified through NDE as being of the poorest quality, did indeed exhibit the lowest performance of the four C/Gl tubes. However, even though this tube contained a large number of defects and broken fibers, it still came very close to or exceeded the NSWC design goals.

Figure 9 shows the set of compressive stress-strain curves for tube 92-91 as supplied by SoRI. Each of the individual curves corresponds to one of the three separate strain gages that were bonded to the tube (the stress-strain curves for the other tubes exhibited similar behavior). The difference in the shape of the curves is attributed to the non-uniform wall thickness of the tube. The curves all exhibit an initial linear region followed by a secondary non-linear region characterized by a steady decrease in slope with increasing strain. The shape of the curves is very similar to that of hot-pressed P-100/BSG flat specimens tested at UTRC. Figure 10 shows the "average" stress-strain curves for all four of the P-100/BSG tubes. Each of these curves was obtained by taking the average of all the strain data for each tube. Again, the same general curve shape (initial linear region followed by a non-linear region) was displayed by all of the tubes.

The results presented in Table 1 indicate that the average performance of the three high quality tubes fell somewhat short of the MSC predicted properties. The measured elastic modulus was fairly close to the predicted value, but the ultimate compressive strength (UCS) was quite a bit below the MSC prediction of 49.5 ksi. One possible explanation for this

discrepancy in strength could be due to the nonuniform wall thickness of the C/Gl tubes (as shown in Figure 6). It is reasonable to assume that compressive buckling most likely initiated at the thinnest section of the tubes, after which ultimate failure rapidly occurred. Recalculating the ultimate compressive strength using a corrected value for cross-sectional area corresponding to the thinnest section of each tube (as determined from the time-of-flight ultrasonic scans) gives an average UCS of 44.1 ksi, which is in much better agreement with the MSC predicted strength.

Also listed in Table 1 are the average compression properties of four different C-C tubes that were previously evaluated on the SACC II program [4]. It is clear that the average performance of the C/Gl tubes is comparable to that of the C-C tubes.

Table 2 provides a comparison of properties for P-100/BSG composites fabricated by hot-pressing and by HIP. Previous comparison of the tensile and compressive performance of another C/Gl composite system (HMU carbon fiber reinforced BSG) indicated no significant difference between hot-pressed and HIP'ed material [5]. The specimens for the unidirectionally reinforced HIP'ed panel were machined from the flat section of a P-100/BSG right-angle bracket that was fabricated using HIP. The results in the Table show that the overall performance of the 0° reinforced composite fabricated via HIP is somewhat lower than that of the 0° reinforced hot-pressed composite. The UCS of the two composites is very similar; however, the compressive elastic modulus of the HIP'ed material is significantly lower than that of the hot-pressed composite. One possible explanation for the poorer performance of the HIP'ed composite could be that some damage may have been done to the high modulus P-100 fibers during processing, most likely during one of the forming operations implemented prior to encapsulation in the HIP can.

For the C/Gl composites with a ply orientation of [0/+20/0/0/-20/0], the HIP'ed tube properties were again lower than those of the hot-pressed panel with respect to both UCS and elastic modulus. Again, it is believed that some damage may have been introduced during one of the forming operations required to make the tube preform in advance of HIP'ing. Fabrication of glass matrix composites using HIP consolidation is still in the early stages. Improvements in HIP processing technique should lead to comparable performance of HIP and hot-pressed composites.

SUMMARY

Continuous P-100 fiber reinforced borosilicate glass tubes were fabricated at UTRC and supplied to SoRI for compression testing. The performance of the C/Gl tubes exceeded the NSWC SACC II program requirements and was comparable to the properties of C-C tubes tested on the SACC II program. This equivalent mechanical performance together with the demonstrated AO resistance [1], thermal damage tolerance [2], high temperature capability (1200°C), and fabricability of C/Gl composites suggests that these C/Gl tubes offer acceptable performance for satellite truss applications.

The properties of the C/Gl tubes were somewhat below those predicted analytically by MSC; however, the discrepancy in compressive strength was resolved by taking into account the non-uniform wall thickness of the tubes. Comparison of the properties of P-100/BSG composites fabricated by hot-pressing and HIP'ing indicated that some damage may have been introduced during the many forming operations that take place to transform the prepregged tape into a composite preform prior to consolidation in the HIP. It is believed that further processing enhancements should lead to improved performance of the C/Gl tubes as more information is gathered on the application of HIP in the processing of glass matrix composite tubes.

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1. W. K. Tredway and K. M. Prewo, "Analysis of the Effect of Space Environmental Exposure on Carbon Fiber Reinforced Glass," UTRC Report R91-112542-4, May 31, 1991.

2. J. McKeever, Oak Ridge National Laboratory, personal communication, 1987.

3. K. M. Prewo, "Carbon Fibre Reinforced Glass Matrix Composite Tension and Flexure Properties," J. Mater. Sci., 23 (1988) 2745-2752.

4. S. Causey, "Fabrication of Carbon-Carbon Tubes, Task V: Characterization and Testing of Components and Sub-Assemblies," Southern Research Institute Presentation at NSWC Satellite Applications for Carbon-Carbon Meeting, Arlington, VA, January, 1991.

5. P. H. McCluskey, W. K. Tredway, and K. M. Prewo, "Fabrication of Carbon Fiber Reinforced Glass Composite Structural Elements," UTRC presentation at the 15th Annual Conference on Composites, Materials, and Structures, Cocoa Beach, FL, January 16-18, 1991.

TABLE 1 - Compression Properties of Carbon Fiber Reinforced Composite Tubes

Tube Type	Specimen #	Area (sq. in.)	Load at Failure (lbs)	Ultimate Compressive Strength (ksi)	Compressive Elastic Modulus (Msi)	Strain to Failure (%)
CARBON FIBER REINFORCED GLASS TUBES						
UTRC C/Gl *(P-100/BSG)*	92-91	0.169	6140	36.2	40.8	0.11
	93-91	0.169	5320	31.6	34.0	0.12
	94-91	0.171	5060	29.6	37.5	0.09
	Ave =	0.170	5507	32.5	37.4	0.11
"Learning Curve" Tube	95-91	0.162	4490	27.8	34.6	0.08
MSC Prediction		0.144	7140	49.5	41.8	
C-C TUBES*						
Kaiser	Ave =	0.148	5235	35.2	30.4	0.13
FMI Braid	Ave =	0.140	3360	24.1	32.7	0.07
FMI Inv.	Ave =	0.193	5988	31.1	33.7	0.10
Hitco Inv.	Ave =	0.120	4544	37.8	50.2	0.08
PROGRAM REQUIREMENTS		min = 0.144 max = 0.169	≥ 4500	≥ 31	≥ 31	

* Ref: S. Causey, "Fabrication of Carbon-Carbon Tubes, Task V: Characterization and Testing of Components and Sub-Assemblies," Southern Research Institute Presentation at NSWC Satellite Applications for Carbon-Carbon Mtg., Arlington, VA, January, 1991.

TABLE 2 - Compression Test Results for P-100/BSG Composites Fabricated by Hot-Pressing and by HIP

FABRICATION METHOD	FIBER VOL%	UCS* (ksi)	ELASTIC MODULUS (Msi)	STRAIN TO FAILURE (%)	SPECIMEN NUMBER
UNIDIRECTIONAL					
Hot-Press (flat panel)	43.2	55.8	51.7	0.53	23-91
HIP (flat panel)	38	53.0	35.0	0.40	312-91
(0/+20/0/0/-20/0)					
Hot Press (flat panel)	38	56.1	44.7	0.48	300-91
HIP (tube)†	37-39	32.5	37.4	0.11	---
Prediction (tube)††	40.0	49.5	41.8	--	---

* Ultimate compressive strength

† Omitting "learning curve" tube data. Average of specimens 92-91, 93-91, and 94-91.

†† Calculations performed by Kent Buesking, Materials Sciences Corporation

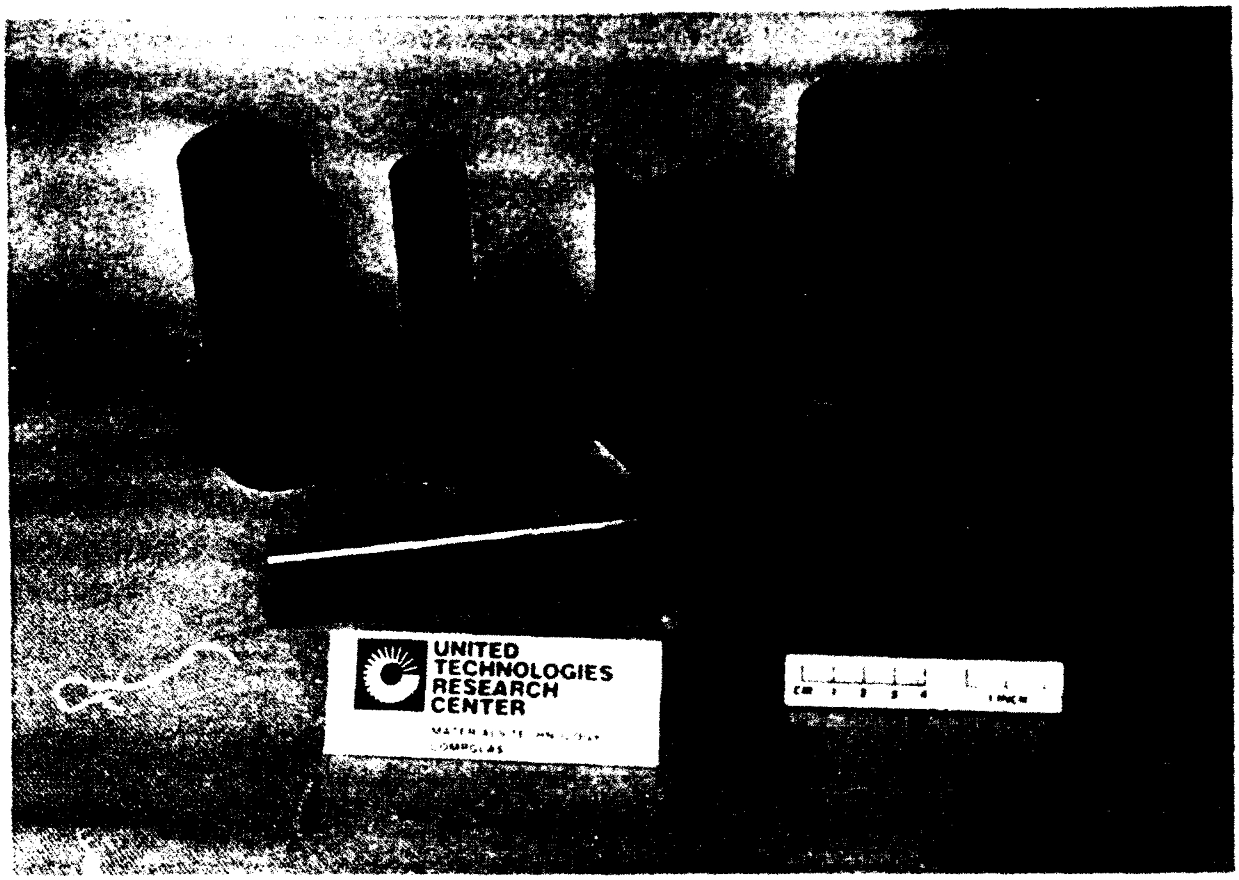

Figure 1 - Continuous carbon fiber reinforced glass tubes and right-angle bracket fabricated using hot isostatic pressing.

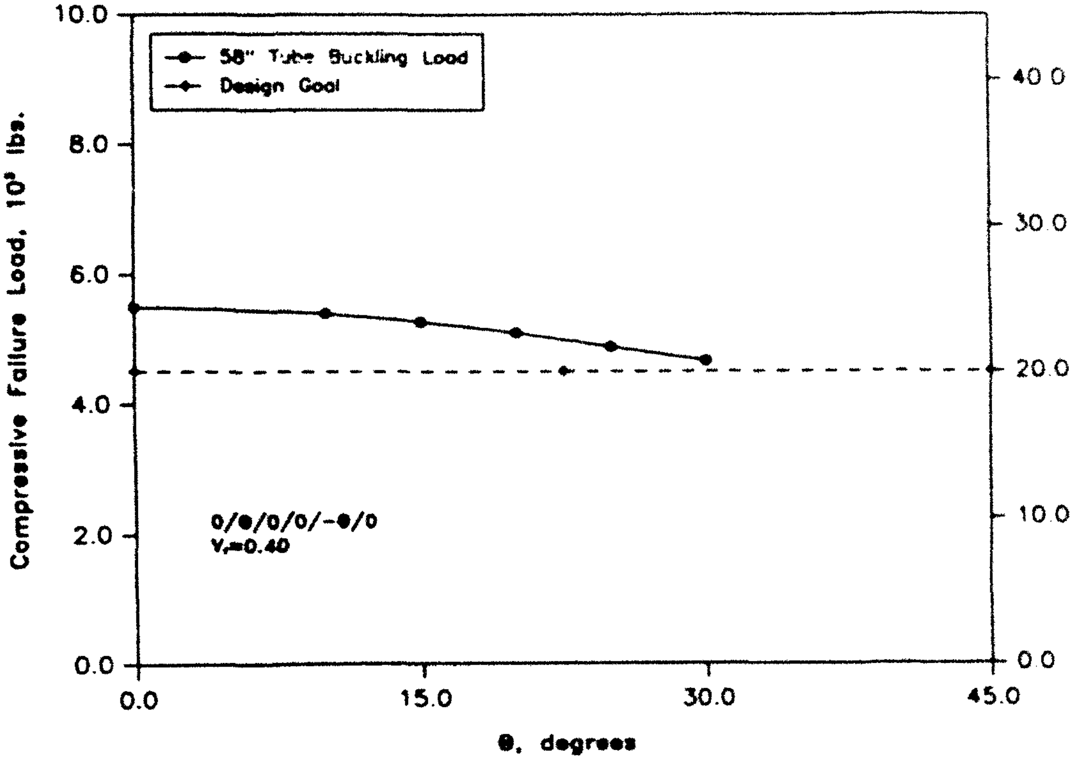

Figure 2 - Calculated compressive load as a function of off-axis ply orientation for a P-100/BSG composite tube (courtesy of Materials Sciences Corporation).

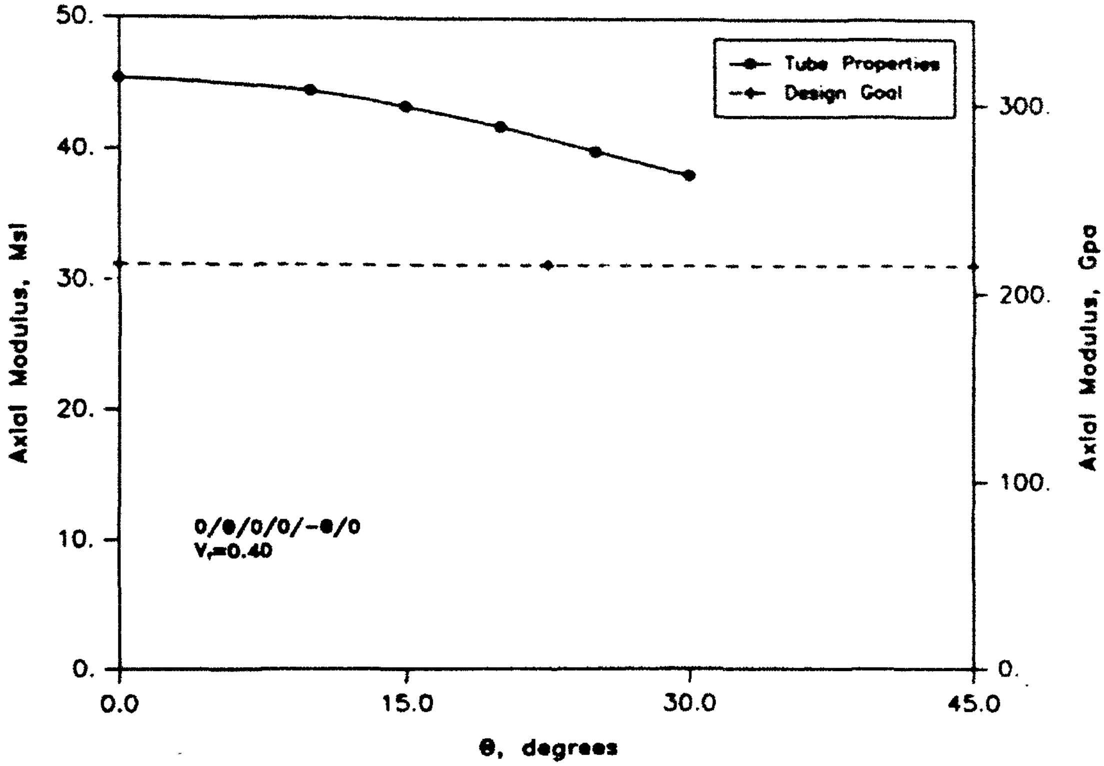

Figure 3 - Calculated compressive elastic modulus as a function of off-axis ply orientation for a P-100/BSG composite tube (courtesy of Materials Sciences Corporation).

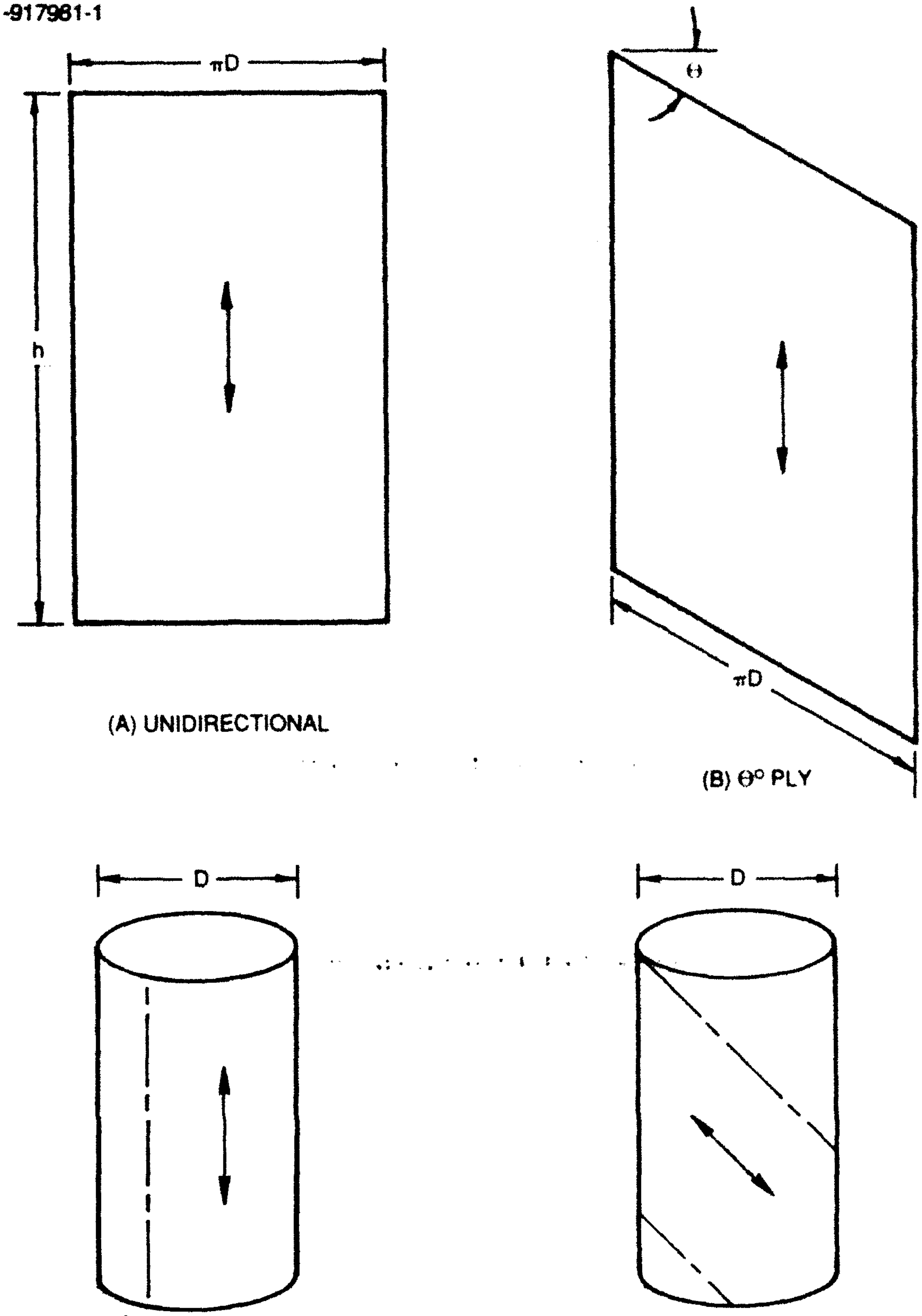

Figure 4: Ply geometry used for tube fabrication. Dashed lines indicate ply seam, and arrows indicate fiber direction.

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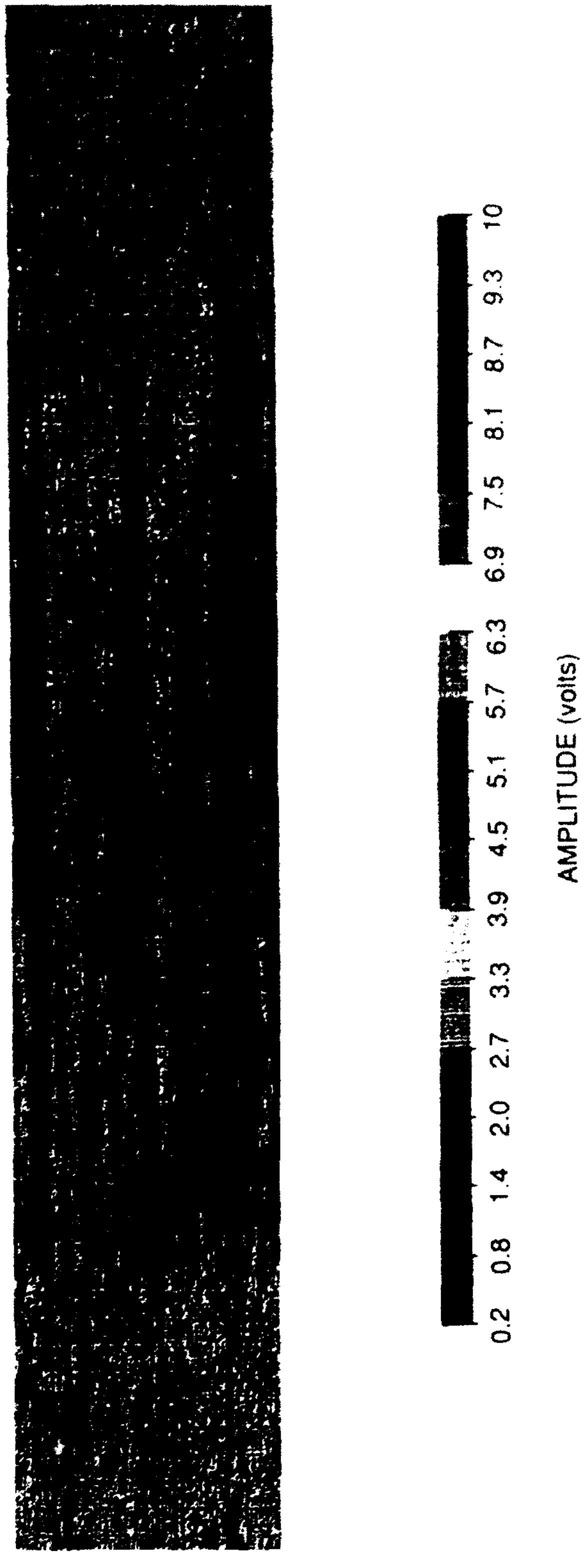

Figure 5 (a): Ultrasonic C-scan of C/GI tube 95-91 showing high attenuation in the central gage section. The attenuated areas correspond to defects in the material. The scan covers the entire circumference of the tube (360°).

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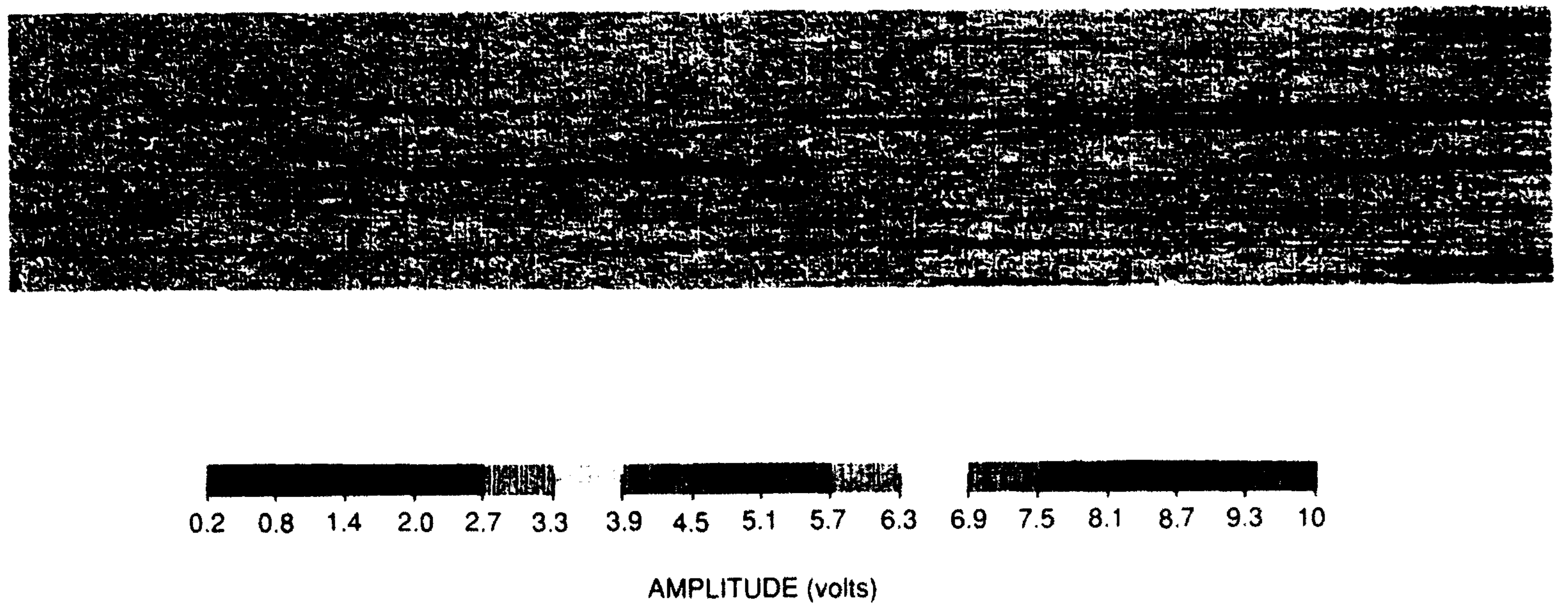

Figure 5(b): Ultrasonic C-scan of C/Gl tube 92-91 showing uniform quality in the central gage section. The scan covers the entire circumference of the tube (360°).

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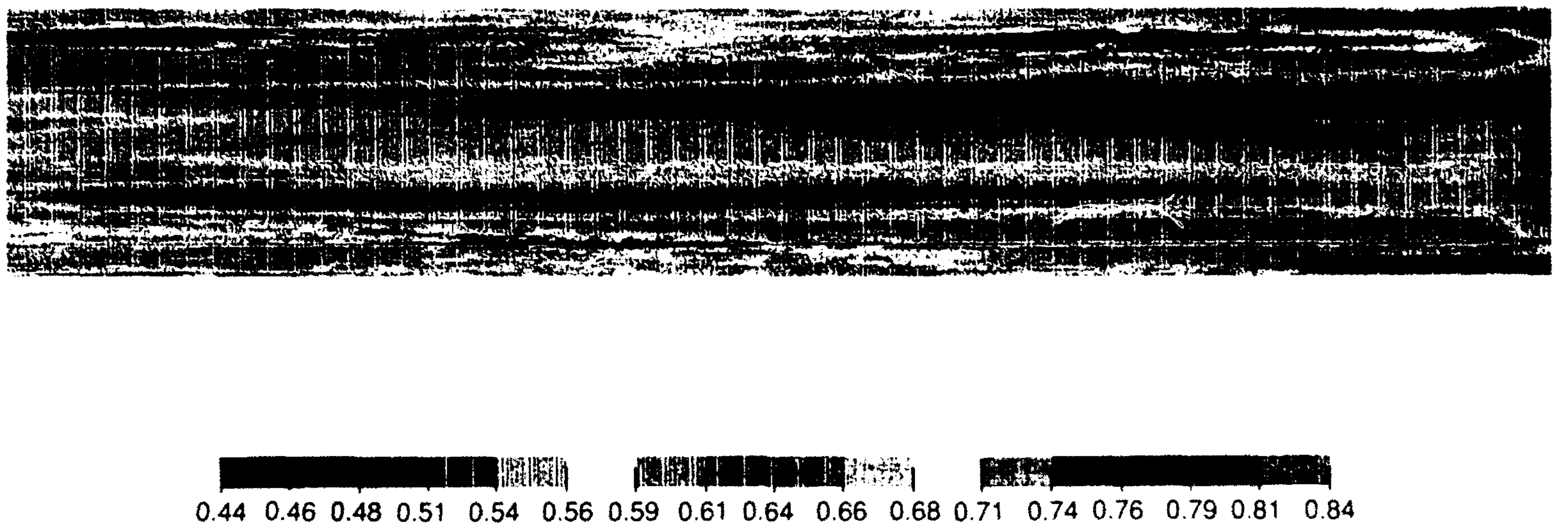

Figure 6: Ultrasonic C-scan (time-of-flight) showing variation in tube thickness around the perimeter of tube 94-91. Wall thickness is fairly constant along the length of the tube but varies circumferentially. The scan covers the entire circumference of the tube (360°).

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Figure 7: X-ray image of C/GI tube 94-91. Image covers approximately one-quarter of the tube (90°).

91-10-4-5

Figure 8: Tube 92-91 after compression testing at SoRI. This tube showed a compressive strength of 36.2 ksi (250 MPa) and an elastic modulus of 40.8 Msi (282 GPa).

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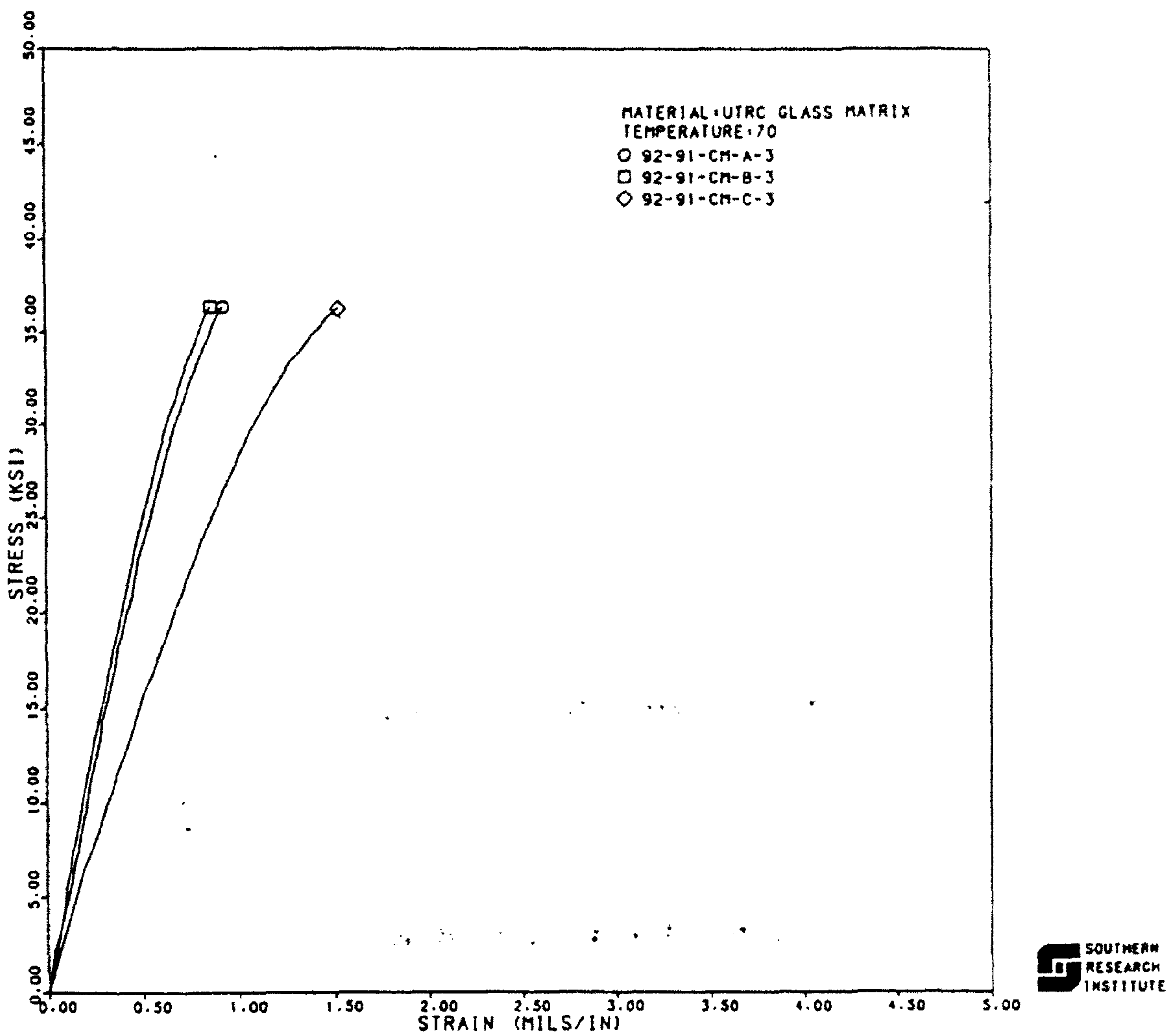

Figure 9 - Compressive stress-strain curves for tube 92-91. Each of the individual curves corresponds to one of the three separate strain gages that were bonded to the tube (curves courtesy of SoRI).

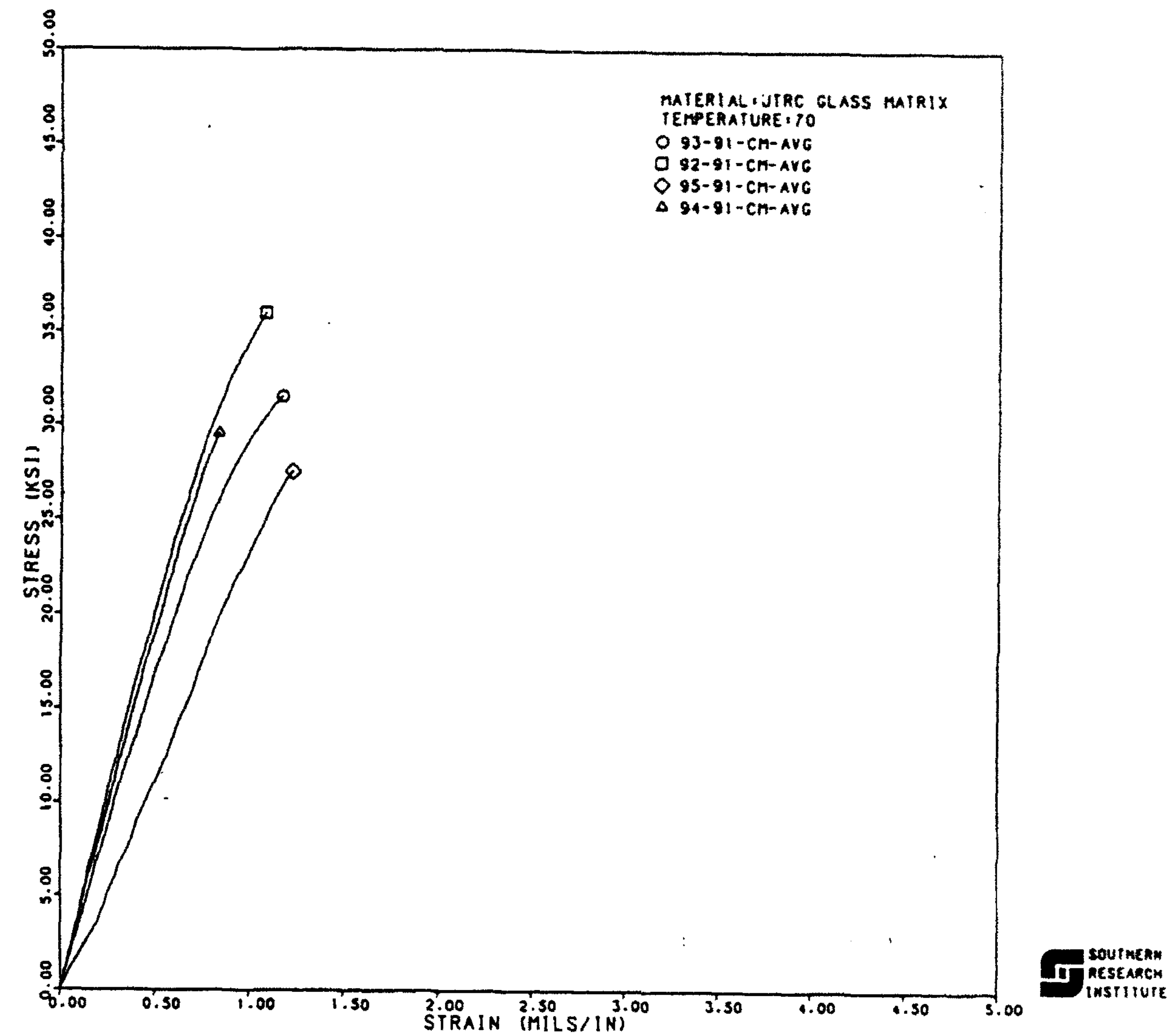

Figure 10 - Average compressive stress-strain curves for the four P-100/BSG tubes. The shape of the curves is very similar to that of hot-pressed P-100/BSG flat specimens tested at UTRC (curves courtesy of SoRI).